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Normal Spectral Emissivity of the Industrially Used Alloys NiCr20TiAl, Inconel 718, X2CrNiMo18-14-3, and Another Austenitic Steel at 684.5 nm

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Abstract Normal spectral emissivity measurements at 684.5 nm for two steels and two nickel-based superalloys at the melting transition and in the molten state are reported in this study. The measurements were performed using a division-of-amplitude photopolarimeter on a microsecond time-scale which is part of a resistive pulse-heating experiment. Emissivity results for the investigated steels do not show any apparent correlation whereas the results for the nickel-based alloys are conclusive and may be used as reference values for similar alloys whenever measurements are too time consuming or not feasible. A weakly increasing emissivity with rising temperature was observed in the liquid state for all the investigated alloys which may be taken into account whenever temperatures are optically measured. However, the measurements presented here show that such a correction does not exceed 0.2 % per 100 K and might only be necessary at a metrological level of accuracy.

Keywords High temperatures · Liquid state · Nickel-based superalloy · Normal spectral emissivity · Steel

1 Introduction

Alloys, especially steels, have a great influence on our everyday lives as they can be found nearly everywhere, for example, in cars, buildings, or even in ball pens. Cur-

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rently, there are more than 3,500 different grades of steel available worldwide [1]. In Europe only, there are about 2,300 standardized steel grades (as of May 2008) registered within the European Steel Registration Office [2]. Although steel is of utmost importance due to its physical properties such as magnetism, acid resistance, or ductility to name a few, it is not possible to investigate all the technical characteristics of every single steel grade as a result of the rapid increase of newly developed brands. Approximately 75 % of currently available steels have been developed over the last 20 years [1], which has led to addition of about 130 new grades every year. For this reason, only major groups of alloys can be characterized concerning their physical properties, and then empirical-based models are deduced for specific steels by major steel producers.

Böhler Edelstahl GmbH & Co KG is one of the world's leading producers of toolsteel and special alloys, and therefore, needs accurate data to maintain or even improve their high quality standard. Required data for several Böhler brand alloys were measured by the Subsecond Thermophysics Group at Graz University of Technology (TU Graz), which by this time has had a long tradition in investigating thermophysical properties of not only pure metals but also of advanced alloys and alloy systems over the past few years. This progress also manifests itself in several cooperation projects with the steel and metal-working industry. Initially, properties like specific enthalpy, electrical resistivity, and thermal expansion (to name a few) were of primary interest for Böhler serving as input data for their in-house simulations of melting and remelting processes. Some of the data measured within this project have also been published in Refs. [3] and [4]. The only parameter that was not investigated so far was the normal spectral emissivity of these alloys due to the laborious measurement procedure. As part of his doctoral research, Wilthan validated the emissivity measurement facility (µs-DOAP) at TU Graz for use with the Fe–Ni system [5]. Prior to this, it was suspected that variations in the alloy composition along the wire-shaped specimens might cause locally dependent emissivity results. Therefore, the DOAP has been exclusively used for measurements on pure metals.

2 Measurements

2.1 Experimental

All measurements of thermophysical properties with molten materials carried out at TU Graz are performed with the so-called ohmic pulse-heating system. The name originates from the discharge of a large pre-stored current pulse over an electrically conducting sample of known initial geometry. In response to this current pulse, a sample can be heated from room temperature to the melting transition and further to the end of the liquid state (boiling point) in about 50 µs to 60 µs (equals heating rates of about $10^8\ \mathrm{K \cdot s^{-1}}$), depending on the material's electrical resistivity.

Any device capable of real-time measuring emissivity of the sample during such a pulse-heating experiment needs to operate at this very same microsecond timescale. Therefore, classical ellipsometric approaches such as using rotating devices (quarter-wave plates and linear polarizers) cannot be used. Instead, a sophisticated

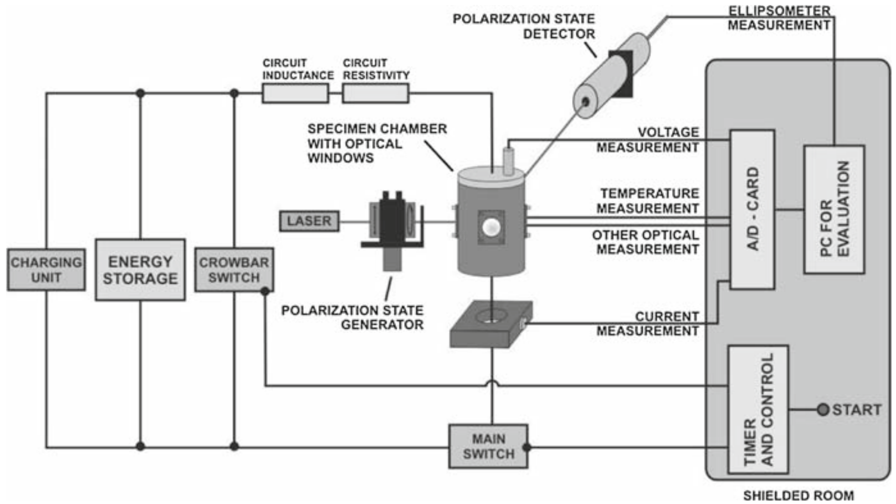

Fig. 1 Functional diagram of the pulse-heating experiment including the µs-DOAP at TU Graz

µs-division-of-amplitude photopolarimeter (DOAP) without any moving parts is used in combination with the standard pulse-heating setup. Figure 1 shows a functional diagram of the experiment. This µs-DOAP, which uses the Stokes formalism for description of the polarization, was already proposed by Azzam [6], but it took another 10 years until Krishnan [7] finally developed and successfully implemented such a polarimeter operating at two different wavelengths in the visible and near-infrared regions.

In short, the change in polarization of an initially linearly (+45°) polarized laser beam is optically detected after reflection from the sample's surface. The reflected beam is collected by an optical system and split into four beams (division-of-amplitude), whose intensities are linked to the four Stokes parameters through the calibration procedure (which yields the instrument's calibration matrix). The Stokes parameters are used to calculate the optical constants n and k and consequently emissivity, ε, of an opaque sample at the given laser wavelength at an angle perpendicular to the sample's surface, thus resulting in the normal spectral emissivity at the used laser wavelength of 684.5 nm. More details and the overall formalism of data reduction have previously been published (see, e.g., [8,9]), and measurement results for optical constants and normal spectral emissivities of several pure metals can be found in Refs. [10] and [11]. The technique has already been successfully used to obtain emissivities of the binary Fe–Ni alloy system [12], and the presently reported measurement results are a continuation of our systematic emissivity investigations with molten materials, extending the DOAP-measurements to more complex alloy systems.

2.2 Specimens

Three of the alloys used in this study were provided by Böhler; the Inconel 718 sample material was originally provided by the National Physical Laboratory (NPL), Teddington, UK, and drawn into wires by Goodfellow Cambridge Limited, UK. These

Table 1 Summary of the chemical composition for the three alloys without copyright-protection

Element	Content (mass%)		
	Nimonic 80A (NiCr20TiAl)	Inconel 718 (NiCr19NbMo)	DIN 1.4435 (X2CrNiMo18-14-3)
Ni	75.6	53.2	14.5
Fe	0.5	18.1	63.2
Cr	19.5	18.6	17.5
Nb	–	5.2	–
Mo	–	3.02	2.7
Ti	2.5	1.01	–
Al	1.7	0.54	–
Mn	–	0.11	1.7
Si	0.16	0.16	0.3
C	0.05	0.03	Max. 0.03
N	–	–	0.07
Zr	0.04	–	–

Data for X2CrNiMo18-14-3 and Nimonic 80A provided by Böhler; Inconel composition taken from [13]

Table 2 Summary of the input parameters for all alloys used during data reduction

	Nimonic 80A	Inconel 718	DIN 1.4435	Austenitic steel (AS)
Sample diameter (mm)	0.60	0.51	0.65	0.70
$T_{solidus}$ (K)	1593	1528	1701	1648
$T_{liquidus}$ (K)	1638	1610	1762	1723

Sample diameters were checked by means of a digital laser micrometer. Temperatures for Inconel 718 are taken from [13], temperatures for the remaining alloys were provided by Böhler

alloys were specifically split into two groups according to their (similar) compositions: austenitic steels or nickel-based alloys. The main idea was to see whether groups of similar alloys would result in comparable emissivity results.

For the Ni-based alloy group, samples of Nimonic 80A and Inconel 718 have been examined, and for the group of ferrous-based austenitic steels, samples of X2CrNiMo18-14-3 and a similar, but copyrighted alloy of undisclosed chemical composition (further denoted as AS) were analyzed. Table 1 lists the chemical compositions of the three known alloys and the respective EN/DIN names of the tested materials.

The samples used for the experiment were wire-shaped (cylindrical) with a nominal length of about 70 mm and diameters ranging from 0.5 mm to 0.7 mm. In order to create a defined initial surface condition, all the samples were treated with abrasive paper (1200 grade) and rinsed with acetone before installation in the discharge chamber. The exact experimental parameters used for each material are listed in Table 2.

2.3 Measurement and Evaluation Procedures

2.3.1 Temperatures

Temperatures reported in this study were measured by means of optical radiation thermometry evaluating the thermal radiation emitted from the sample surface. Owing to the moderate visible radiation emitted at typical melting temperatures of steels and alloys, a pyrometer operating in the near-infrared at a center wavelength of 1570 nm (bandwidth of 84 nm FWHM) and f/2.4 optics had to be used. This unavoidable choice of a NIR-pyrometer limits the accuracy of the evaluated temperature as the obtained emissivities could not be used to improve the pyrometrically obtained temperatures. The investigated alloys would have to be gray bodies (constant emissivity with wavelength, but neither values of 0 or 1) with respect to emissivity to allow the linkage of temperatures with measured emissivity.

Therefore, reported temperatures were evaluated from the recorded radiances by in situ calibration of the pyrometer for each experiment at a known temperature, in our case the arithmetic mean of solidus and liquidus temperatures. This choice was found to be the most convenient for alloys with the main drawback that a possible change in emissivity is not accounted for and assumed to be constant, instead, throughout the liquid state. More information addressing this issue can be found in Sect. “4”. For lack of emissivity measurement capabilities in the solid, the same constant emissivity obtained at the liquidus temperature was also used in the solid state to convert the measured radiance to a (true) temperature.

2.3.2 Uncertainties

At the Subsecond Thermophysics Group at TU Graz, we decided to follow international accredited guidelines and usually report all uncertainties evaluated according to the concept of GUM [14]. Normal spectral emissivity marks an exception as a detailed analysis leads to expanded (coverage factor of 2) uncertainties for alloys of about 160 % [5]. Comparisons of measurement results with available literature values indicate much better agreement than one would expect by looking at the previusly mentioned number. Another indication of the inapplicability of the GUM concept for the emissivity setup comes from the statistical analysis of individual emissivity measurements: neither reproducibility nor standard deviation support such large uncertainty estimates. The GUM algorithm fails due to missing information about the installed A/D measurement board (obsolete and no longer supported) in the data acquisition system. Therefore, the scatter of the individual measurements in combination with values from other investigated materials and experience are used instead to obtain an uncertainty estimate. The recent results are similar to the situation reported in Ref. [5], and therefore the same uncertainty estimates will be used. Absolute uncertainties are 0.03 for all the investigated alloys with the exception of 0.07 for X2CrNiMo18-14-3, where not only fewer measurements have been performed but also a larger scatter was observed. Relative uncertainties depend on the actual emissivity values but are, e.g., on the order of 8 % for the nickel-based alloys.

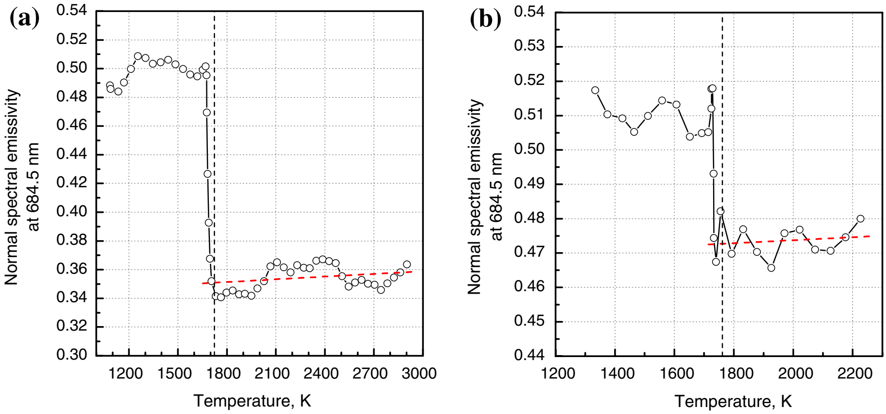

Fig. 2 Normal spectral emissivity at 684.5 nm of two steels as a function of temperature: (a) an (undisclosed) austenitic steel and (b) X2CrNiMo18-14-3. *Open circles* average of several DOAP measurements, *dotted line* linear least-squares fit to the liquid state, *vertical dashed line* liquidus temperature

3 Results

A set of 7–10 individual experiments was conducted for each of the investigated alloys and the results were averaged to reduce some of the random scatter of the DOAP system. The only exception is marked by the X2CrNiMo18-14-3 alloy, where only three measurements have been averaged as the rest of the available sample material had to be used for thermophysical property measurements. Unfortunately, we had no chance to obtain a new batch of X2CrNiMo18-14-3 in time for this investigation.

Results for all the four investigated alloys are graphically presented as emissivity versus temperature traces as shown in Figs. 2 and 3. In the liquid phase, the emissivity has been approximated by linear least-squares fits, as for any other more complex liquid state behavior, like the one mentioned by the authors of Ref. [15], and is neither expected nor yet proven. Any structure in the measurement results (see, e.g., Figs. 2a and 3a) is to the best of our knowledge purely random and can only be used to quantify realistic uncertainty estimates. The respective fit parameters, the temperature range of validity of those fits, and further important results are, for convenience, summarized in Table 3.

4 Discussion

The applicability of the μs-DOAP for emissivity measurements of binary alloys has already been tested and demonstrated in Refs. [5] and [12]. Therefore, the primary focus of this investigation was not on seeing whether this technique would also work for more complex alloys but rather to check if a common trend in emissivity can be found for similar (composition-wise) alloys or groups of alloys such as nickel-based alloys. As already stated in the introductory comments, too many new alloys (even more than 100 grades of steel alone each year) are developed and put on the market which makes a detailed characterization of each single material not feasible. In

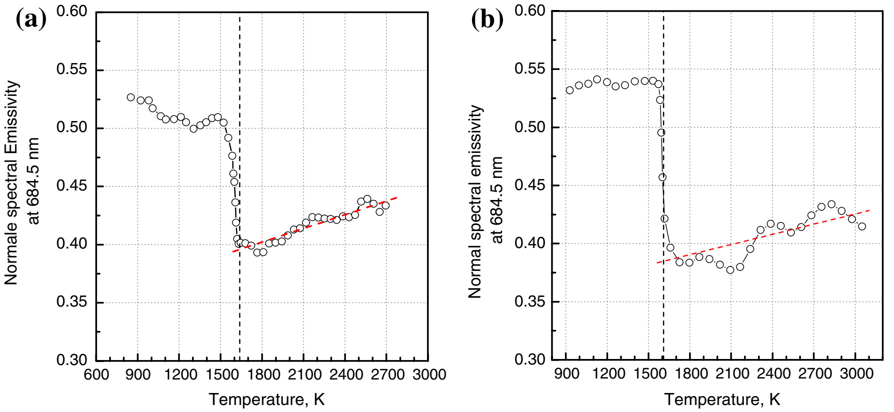

Fig. 3 Normal spectral emissivity at 684.5 nm of two nickel-based alloys as a function of temperature: (**a**) Nimonic 80A and (**b**) Inconel 718. *Open circles* average of several DOAP measurements, *dotted line* linear least-squares fit to the liquid state, *vertical dashed line* liquidus temperature

Table 3 Summary of the evaluated data for the four investigated alloys

Material	ε at T_{liq}	Linear fit coefficients		T-range (K)	ε at T_{max}	$\Delta\varepsilon$ $(\% \cdot (100\,K)^{-1})$
		a	b			
Nimonic 80A	0.395	0.331	39.30×10^{-6}	1638–2700	0.437	0.99
Inconel 718	0.385	0.338	29.23×10^{-6}	1610–3050	0.427	0.76
X2CrNiMo18-14-3	0.473	0.465	4.36×10^{-6}	1762–2250	0.475	0.09
Austenitic steel	0.351	0.340	6.29×10^{-6}	1723–2900	0.358	0.18

The fit results are given by the coefficients of a linear least-squares fit according to $\varepsilon(T) = a + bT$, where T is the temperature as stated in the column T-range individually for each material. ε at $T_{liq.}$ and ε at T_{max} are the calculated emissivity values at the liquidus and maximum temperatures, respectively, and $\Delta\varepsilon$ is the relative change in the liquid state emissivity in % per 100 K

order to shorten the time consumed in the task of measuring each alloy individually, it would be rather helpful to have some rules of thumb at hand to estimate such intricate properties as emissivity.

4.1 Comparison of Emissivities

4.1.1 Steels

The first test group consisted of the two austenitic steels. Although the exact composition of AS had to remain undisclosed due to copyright reasons, it was picked because of its similar content of iron compared to X2CrNiMo18-14-3. Just by visually inspecting the plots shown in Figs. 2a and b one can see that besides the common behavior of a dropping emissivity at the melting transition partly due to smoothing of the surface (as a result of the surface tension), there are no real other similarities for X2CrNiMo18-14-3 and AS. Both steels do show an insignificant increase in emissivity of maximum 0.18 % $\cdot$ (100 K)$^{-1}$ for AS and 0.09 % $\cdot$ (100 K)$^{-1}$ for X2CrNiMo18-14-3 in the liquid state for the investigated temperatures. The absolute emissivity values at the liquidus temperature are 0.350 for AS and 0.473 for X2CrNiMo18-14-3, making X2CrNiMo18-14-3 the highest liquid state emissivity detected with our system to date. Previous measurements for pure iron yielded at melting $\varepsilon = 0.362$ (with an increase of 0.84 % $\cdot$ (100 K)$^{-1}$) and a binary Fe62.7Ni mass% alloy (iron content comparable with X2CrNiMo18-14-3) yielded $\varepsilon = 0.295$ (with an increase of 2.72 % $\cdot$ (100 K)$^{-1}$) at T_{liq} [12]].

These reported numbers demonstrate the challenges of attempting to give an estimated value for the emissivity of steels and alloys in general. Although X2CrNiMo18-14-3 mostly consists of iron, its emissivity differs significantly from pure metallic iron or that of the Fe62.7Ni and AS alloys. As our results indicated so far, this is mostly due to the complex phase diagram of steel, the diverse production processes, and the changes in physical properties induced by only a small amount of an added or altered component.

4.1.2 Nickel-Based Alloys

A look at the second test group consisting of Nimonic 80A and Inconel 718 (see Fig. 3a, b) indicates a closer relation between these two alloys. The emissivity results for both materials are comparable to each other in two aspects: (i) closely matched emissivity values at T_{liq}, and (ii) similar liquid state emissivity change with temperature. At the respective liquidus temperatures, Nimonic 80A yields $\varepsilon = 0.395$ (with an increase of 0.99 % $\cdot$ (100 K)$^{-1}$) and Inconel yields $\varepsilon = 0.385$ (with an increase of 0.76 % $\cdot$ (100 K)$^{-1}$). For both, the emissivity increase in the liquid state of about 1 % $\cdot$ (100 K)$^{-1}$ is not very significant, but the low scatter detected for Nimonic 80A indicates that the effect is real and does not solely originate from the statistical treatment of the raw data. Previously published emissivity results [12] at melting for pure nickel yielded $\varepsilon = 0.365$ (with an increase of 0.31 % $\cdot$ (100 K)$^{-1}$). Other recent emissivity investigations with a ternary NiCrSi (Si content about 1.5 mass%) alloy are also in agreement with the results presented herein and will be published in the near future [16].

Contrary to the investigated types of steel and based on the findings of our measurements, it seems more appropriate to give a liquid-state estimate for the emissivity of nickel-based alloys. Numerous measurements with both pure metals and alloys led to the conclusion that alloys usually have higher emissivities at T_{liq} than the respective dominant constituent in its pure metallic form at melting. As can be seen, this holds for the investigated nickel-based alloys when comparing the results with the emissivity of pure nickel.

4.2 Effect of Emissivity on Measured Temperature

Finally, we have to deal with the pending question as to what extent does the changing emissivity influence the optical temperature measurement. As mentioned before (see Sect. 2.3.1), all the temperatures reported in this study were measured by an optical pyrometer and evaluated using a constant emissivity for temperatures above the melting transition. The actual measured emissivities could not be used directly to improve temperature evaluation as there is a wavelength mismatch (1570 nm for the pyrometer and 684.5 nm for the DOAP) and a strict gray-body behavior cannot be presumed for alloys in the visible to near-infrared regions. As a result, all the temperatures are inaccurate by a certain amount as the emissivity changes. These changes in emissivity as stated before and given in Table 3 can be converted to temperature uncertainties assuming temperatures were also measured at or in the vicinity of 684.5 nm (the standard wavelength in pyrometry is 650 nm, for which this estimation should still be valid). As the emissivity increases throughout the liquid state for all the investigated alloys, calculated temperatures are too high.

For the two steels, X2CrNiMo18-14-3 and AS, the relative correction in temperature at 684.5 nm (due to the changing emissivity) would be $-0.01\,\% \cdot (100\,\mathrm{K})^{-1}$ (difference of -1 K at the maximum temperature stated in Table 3) and $-0.04\,\% \cdot (100\,\mathrm{K})^{-1}$ (difference of -9.1 K at $T_{\max}$), respectively.

The same calculation for the nickel-based alloys yields a correction in temperature at 684.5 nm of $-0.20\,\% \cdot (100\,\mathrm{K})^{-1}$ (difference of -34.5 K at $T_{\max}$) for Nimonic 80A and $-0.19\,\% \cdot (100\,\mathrm{K})^{-1}$ (difference of -45.1 K at $T_{\max}$) for Inconel 718.

While a temperature uncertainty of about 45 K at 3050 K might be good enough for several industrial applications or as estimates for computer-based simulations of processes, it will certainly be too low for metrological investigations. Therefore, when it comes to accuracy in temperature measurement, the undertaken experimental efforts strongly depend on the actual application.

5 Conclusion

Normal spectral emissivity measurements at 684.5 nm using a DOAP on a microsecond time scale have been performed for the X2CrNiMo18-14-3 and another austenitic steel as well as for the two nickel-based alloys, Nimonic 80A and Inconel 718. The summarized measurement results for all the four alloys can be found in Table 3. In the liquid state, all the investigated materials show a slight increase of emissivity with temperature, a behavior that was previously observed for other alloys as well. It is not understood why almost all the alloys show increasing emissivities, whereas constant or decreasing emissivity behaviors in the liquid state have been observed and reported for pure metals [9]. However, the change of emissivity with temperature is weak, and therefore a correction of temperatures in the liquid state (when using optical pyrometers operating in the visible or near-infrared regions) for most industrial applications is not required.

Comparison of the results for the two different grades of steels leads to the conclusion that a rule of thumb does most likely not exist to estimate the liquid emissivity. A different situation occurred for the nickel-based alloys: so far, all the results indicate good agreement between different nickel alloys and the usage of the reported results as best estimate seems feasible.

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